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## 2.4 - Química do solo

# NEW METHODS TO QUANTIFY $\text{NH}_3$ VOLATILIZATION FROM FERTILIZED SURFACE SOIL WITH UREA<sup>(1)</sup>

Ana Carolina Alves<sup>(2)</sup>, Patrícia Perondi Anchão Oliveira<sup>(3)</sup>, Valdo Rodrigues Herling<sup>(4)</sup>, Paulo Cesar Ocheuze Trivelin<sup>(5)</sup>, Pedro Henrique de Cerqueira Luz<sup>(4)</sup>, Teresa Cristina Alves<sup>(3)</sup>, Ramon Cellin Rochetti<sup>(6)</sup> & Waldomiro Barioni Júnior<sup>(3)</sup>

## SUMMARY

Gaseous N losses from soil are considerable, resulting mostly from ammonia volatilization linked to agricultural activities such as pasture fertilization. The use of simple and accessible measurement methods of such losses is fundamental in the evaluation of the N cycle in agricultural systems. The purpose of this study was to evaluate quantification methods of  $\text{NH}_3$  volatilization from fertilized surface soil with urea, with minimal influence on the volatilization processes. The greenhouse experiment was arranged in a completely randomized design with 13 treatments and five replications, with the following treatments: (1) Polyurethane foam (density  $20 \text{ kg m}^{-3}$ ) with phosphoric acid solution absorber (foam absorber), installed 1, 5, 10 and 20 cm above the soil surface; (2) Paper filter with sulfuric acid solution absorber (paper absorber, 1, 5, 10 and 20 cm above the soil surface); (3) Sulfuric acid solution absorber (1, 5 and 10 cm above the soil surface); (4) Semi-open static collector; (5)  $^{15}\text{N}$  balance (control). The foam absorber placed 1 cm above the soil surface estimated the real daily rate of loss and accumulated loss of  $\text{NH}_3\text{-N}$  and proved efficient in capturing  $\text{NH}_3$  volatilized from urea-treated soil. The

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<sup>(2)</sup> Professor, State University of Mato Grosso do Sul, Route MS 306, Km 6, CEP 79450-000 Cassilândia (MS). E-mail: anacarolina@uems.br

<sup>(3)</sup> Researcher, EMBRAPA/CPPSE. Post Office Box 339, CEP 13560-970 São Carlos (SP). E-mails: ppaolive@cnpse.embrapa.br; teresa@cnpse.embrapa.br; barioni@cnpse.embrapa.br

<sup>(4)</sup> Professor, Department of Animal Science, USP/FZEA. Post Office Box 23, CEP 13630-970 Pirassununga (SP). E-mails: vrherling@usp.br; phcerluz@usp.br

<sup>(5)</sup> Professor, University of São Paulo – USP. Center of Nuclear Energy in <sup>(5)</sup> Agriculture – CENA. Post Office Box 96, CEP 13400-970 Piracicaba (SP). E-mail: pcotrive@cena.usp.br

<sup>(6)</sup> Graduate student in Animal Science, Statel University Paulista – UNESP. Campus de Ilha Solteira. Post Office Box 31, CEP 15385-970 Ilha Solteira (SP).

estimates based on acid absorbers 1, 5 and 10 cm above the soil surface and paper absorbers 1 and 5 cm above the soil surface were only realistic for accumulated N-NH<sub>3</sub> losses. Foam absorbers can be indicated to quantify accumulated and daily rates of NH<sub>3</sub> volatilization losses similarly to an open static chamber, making calibration equations or correction factors unnecessary.

**Index terms:** fertilizer-N, ammonia absorber, <sup>15</sup>N, NH<sub>3</sub>-N loss.

## RESUMO: MÉTODOS PARA QUANTIFICAÇÃO DA VOLATILIZAÇÃO DE AMÔNIA EM SOLO FERTILIZADO NA SUPERFÍCIE COM UREIA

*As perdas gasosas de nitrogênio do solo são consideráveis, sendo resultantes principalmente da volatilização de amônia ligada a diversas atividades agropecuárias, como na adubação de pastagens. A utilização de métodos simples e acessíveis para mensurar essas perdas é fundamental na avaliação do ciclo de N nesses sistemas. Assim, o objetivo desta pesquisa foi avaliar métodos usados para quantificar a NH<sub>3</sub> volatilizada da ureia aplicada na superfície do solo, considerando fundamental que tenham mínima interferência no processo de volatilização. O experimento foi conduzido em casa de vegetação, utilizando-se um delineamento experimental inteiramente casualizado, com 13 tratamentos e cinco repetições. Os tratamentos foram: (1) absorvedor de espuma de poliuretano (densidade de 20 kg m<sup>-3</sup>) contendo solução de ácido fosfórico e colocado a 1, 5, 10 e 20 cm acima da superfície do solo; (2) absorvedor de papel-filtro contendo solução de ácido sulfúrico e colocado a 1, 5, 10 e 20 cm acima da superfície do solo; (3) absorvedor com solução de ácido sulfúrico e colocado a 1, 5 e 10 cm acima da superfície do solo; (4) coletor semiaberto estático; e (5) balanço de <sup>15</sup>N (método de referência). O absorvedor de espuma colocado a 1 cm acima da superfície do solo indicou valores reais para a taxa diária de perda e a perda acumulada de N-NH<sub>3</sub>, sendo eficiente na captação da NH<sub>3</sub> volatilizada da ureia aplicada ao solo. Os absorvedores com ácido sulfúrico nas alturas de 1, 5 e 10 cm do solo e os absorvedores de papel nas alturas de 1 e 5 cm do solo apresentaram estimativas reais somente das perdas acumuladas de N-NH<sub>3</sub>. O absorvedor de espuma é uma câmara estática aberta indicada para quantificar a taxa de perda diária ou a perda acumulada de amônia por volatilização, não sendo necessário o uso de equações de calibração ou fatores de correção.*

*Termos de indexação:* fertilizante nitrogenado, absorvedor de amônia, <sup>15</sup>N, perdas de N-NH<sub>3</sub>.

## INTRODUCTION

Pastures constitute the basis of beef cattle production in Brazil. Economically, production systems that use this feed source are highly competitive in comparison with systems based on agroindustrial by-products, cereal grains and silage (Hodgson, 1990). Fertilization is considered the best alternative to increase beef production, since the possibility of increasing pasture area is limited (Balsalobre, 2002).

For the economic viability of pasture-based beef production it is essential to know how to use fertilizers appropriately to intensify pasture productivity. Nowadays, the maximum efficiency of a fertilizer must be directly related to the lowest environmental impact. Nitrogen fertilizers are the most commonly used, the most expensive and have the greatest potential to damage the environment of all fertilizers.

Gaseous N losses are the main cause of inefficiency of N-fertilizers, contributing to the emission of

ammonia, molecular N (N<sub>2</sub>) and N oxides, mainly nitrous oxide, into the atmosphere. Such losses must be understood for an appropriate pasture fertilization management and to rationalize fertilizer use. However, methods used to estimate ammonia volatilization losses are expensive and may alter the environmental conditions near the soil surface, by the presence of a collection chamber, and bias the results (Lara-Cabezas & Trivelin, 1999; Araújo et al., 2009). Due to these difficulties and the high cost of NH<sub>3</sub>-N losses from pasture systems (Oliveira, 2001; Oliveira et al., 2003, 2007), simple and accessible methods must be developed to evaluate these processes, which are also essential to analyze the N cycle. New methods for the estimation of ammonia volatilization must be calibrated using the <sup>15</sup>N isotope balance technique as reference (Lara Cabeza et al., 1999; Araújo et al., 2009). Thus, the objective of this study was to evaluate and calibrate new quantification methods of N-NH<sub>3</sub> volatilized from urea-treated soil, with minimal disturbance of the volatilization processes.

## MATERIAL AND METHODS

The experiment was carried out in a greenhouse of the Department of Animal Science/College of Animal Science and Food Engineering, USP, Pirassununga, SP (21° 59' S, 47° 26' W, 634 m asl).

Soil was sampled in the 0–20 cm layer of a Typic Hapludox (Embrapa, 1999). Soil bulk density was measured by the volumetric ring method (Machado et al., 2005). Lime analysis was performed according to the saturation base method (Quaggio & Raij, 1996), after which soil samples remained incubated during 90 days. At the end of the incubation period the chemical analysis (Machado et al., 2005) indicated pH ( $\text{CaCl}_2$ ) of 6.6; K, Ca and Mg concentrations of 1.7, 39 and 14  $\text{mmol}_c \text{dm}^{-3}$ , respectively; CTC of 63  $\text{mmol}_c \text{dm}^{-3}$ ; and base saturation (V %) of 87 %. Temperature and relative humidity means in the greenhouse are presented in figure 1.

The experiment was arranged in a completely randomized design with 13 treatments and five replications. The factors studied were methods of  $\text{NH}_3$ -loss evaluation and distances between the soil surface and  $\text{NH}_3$ -absorbers, by the following methods: (1) Polyurethane foam (density of 20  $\text{kg m}^{-3}$ ) with phosphoric acid solution absorber (foam absorber) placed 1, 5, 10 and 20 cm above the soil surface; (2) Paper filter with sulfuric acid solution absorber (paper absorber, 1, 5, 10 and 20 cm above the soil surface); (3) Sulfuric acid solution absorber (1, 5 and 10 cm above the soil surface); (4) Semi-open static collector and (5)  $^{15}\text{N}$  balance as reference method.

The experiment lasted from August 26 to September 15, 2005. Plastic pots (17 x 20 cm) without holes for water drainage, to avoid N loss by leaching were filled with 4.4 kg of finely ground air-dried soil. Each pot represented a plot and was fertilized with N at a rate corresponding to 100  $\text{kg ha}^{-1}$  N-urea. The pots for the foam, paper absorber and semi-open static collector treatments were fertilized with normal urea, while those for the  $^{15}\text{N}$  balance treatment received enriched urea (5 %  $^{15}\text{N}$ ). The fertilizer was diluted in water and then applied to the soil surface. Absorbers

and collectors were placed above the pots according to the treatments, except for the  $^{15}\text{N}$  balance treatment, where no absorber or collector was installed.

The foam and paper filters of the absorbers and collectors were changed every two days to determine the volatilized ammonia, totaling 11 samples in a period of 22 days.

The pots placed on benches were protected by transparent plastic curtains to avoid interference of the air flow from the greenhouse exhaust fans with the capture of volatilized ammonia. Soil moisture was maintained at 50 % of the available water content (AWC), approximately. Pots were daily weighed and watered as required.

The foam absorbers (described in Figure 2) consisted of polyurethane (PU) foams (8.0 x 8.0 cm; density 20  $\text{kg m}^{-3}$ ) soaked with 11 mL of phosphoric acid solution (0.167  $\text{mol L}^{-1}$ ). They were placed above PVC plates (10.0 x 10.0 x 0.2 cm) and wrapped in a layer of polytetrafluoroethylene tape (PTFE, density 0.25–0.35  $\text{g cm}^{-3}$ , thickness 0.05–0.06 mm), ammonia-permeable and water impermeable. The plates were then installed upside-down above the pots, supported by four plastic rods, maintaining the absorbers at the established distances from the soil surface.

For the paper absorbers, filter paper (8.0 x 8.0 cm) was immersed in 2 mL of  $\text{H}_2\text{SO}_4$  solution (5  $\text{mol L}^{-1}$ ) and the collectors were installed like the foam absorbers.

To prevent contamination with environmental  $\text{NH}_3$ , the absorbers were maintained in plastic bags until installation on the pots. Above each pot one absorber was installed at a specific height, according to the treatment (1, 5, 10 or 20 cm). The PVC plates on the upper side of the absorbers prevented the capture of ammonia present over the collector. After two days, the absorbers were removed, deep-frozen ( $-18^\circ\text{C}$ ) in plastic bags and immediately replaced by new absorbers.

At the end of the experimental period, the absorbers were disassembled and components washed with deionized or distilled water. For each absorber, the

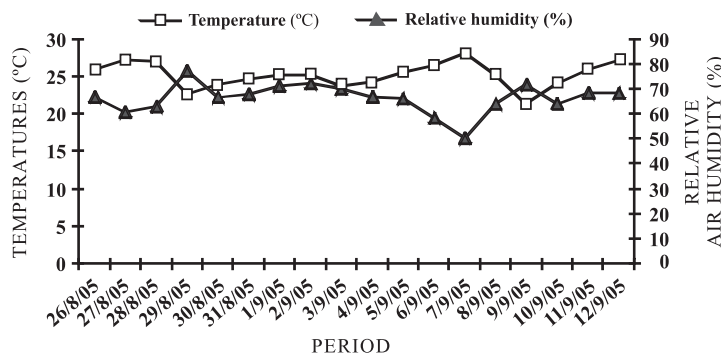
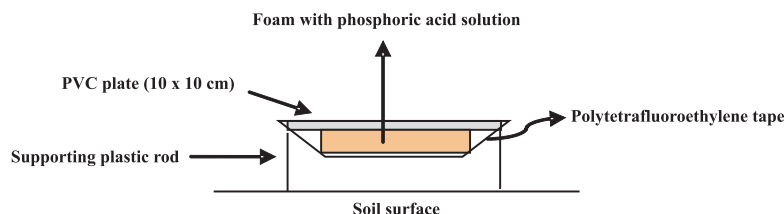


Figure 1. Mean temperatures (°C) and relative air humidity (%) during the experimental period (2005).



**Figure 2. Foam absorbers.**

PVC plate and the PTFE tape were washed with approximately 30 mL of water, using a wash bottle, above the PU foam placed in a Büchner funnel attached to a Kitassato and a vacuum pump. For a complete removal of the acid solution, the foam was washed a second time with distilled or deionized water (approximately 300 mL) in the Büchner-Kitassato apparatus. The solution collected in the Kitassato was then transferred to a Backer and the volume recorded for later calculations. To determine N, 50 mL aliquots were analyzed by the steam distillation and titration micro-Kjeldahl procedure (Carmo et al., 2005). For the paper absorbers, each paper filter was finely ground in a mortar and mixed in a Backer with the water used to wash the PVC plate and the PTFE tape (approximately 150 mL). A 50 mL aliquot of the solution produced for each absorber was analyzed for N by the micro-Kjeldahl procedure.

The acid solution absorbers consisted of 35 mL plastic containers (3.5 cm diameter) filled with 5 mL of  $\text{H}_2\text{SO}_4$  solution ( $5 \text{ mol L}^{-1}$ ). The top of each container was sealed with PTFE tape (same type as described before) fixed in place by the container's lid in which a 3 cm in diameter hole was previously produced. The absorbers were maintained in plastic bags until transference to the plots. The absorbers were fixed upside-down on plastic rods at the established distances from the soil surface. After two days the absorbers were replaced by new ones. The liquid from the containers were placed in sealed and labeled flasks and deep-frozen. To quantify the absorbed N, the flask content was diluted in water to 150 mL. Samples (50 mL) were separated for the micro-Kjeldahl procedure.

The semi-open static collectors used in the experiment had been developed by Nönmik (1973), and adapted by Lara Cabezas et al. (1999). The PU foam pieces were soaked in 30 mL of phosphoric acid solution ( $0.167 \text{ mol L}^{-1}$ ) with 5 % glycerin and stored in plastic bags before being placed in the collectors. Foams were removed and replaced every two days until the 22<sup>nd</sup> day. The foams located on the upper part of the collectors were discarded and those located on the lower part were put in plastic bags, identified and deep-frozen until analysis. To quantify N, each foam was washed with water (400 mL, approximately) in a Büchner-Kitassato vacuum pump system and a 50 mL sample was separated for the micro-Kjeldahl procedure. Due to the usual variations in internal conditions in semi-open static collectors (increase in

soil moisture and recycling of vaporized water), which interfere with the natural volatilization process (Lara Cabezas & Trivelin, 1990), calibration equations (Lara Cabezas et al., 1999) were used to express  $\text{N-NH}_3$  volatilization.

For the  $^{15}\text{N}$  balance treatment, N was applied at a rate of  $100 \text{ kg ha}^{-1}$  N of enriched urea (5 atom %  $^{15}\text{N}$ ). After 22 days, samples were taken from each pot, from three layers (0–5, 5–10 and 10–15 cm). A  $\text{H}_2\text{SO}_4$   $5 \text{ mol L}^{-1}$  solution was sprayed on the surface of each sample, to avoid  $\text{NH}_3$  volatilization, and the soil dried in a forced-air oven ( $40^\circ\text{C}/72 \text{ h}$ ) for posterior analysis by mass spectrometry (ANCA SL) at the CENA/USP laboratory. The non-enriched soil was also evaluated for natural  $^{15}\text{N}$  abundance.

To estimate ammonia volatilization losses, by the  $^{15}\text{N}$  balance method, the following aspects were evaluated:

1. N content ( $\text{g kg}^{-1}$ ) and  $^{15}\text{N}$  content (atom %) in the soil from the plots – quantified in a mass spectrometer attached to an ANCA-SL analyzer (Europa Scientific Ltda.);
2. N percentage originated by the fertilizer (%SNFF) in the soil, according to the equation:  $\% \text{SNFF} = [(a-c)/(b-c)] \times 100$ , where:  
 $a = ^{15}\text{N}$  abundance in the soil (atoms %)  
 $b = ^{15}\text{N}$  abundance in the labeled fertilizer  
 $c = ^{15}\text{N}$  natural abundance in the control soil (without fertilizer)
3. Amount of soil N derived from the fertilizer (ASNFF), according to the equation:  
 $\text{ASNFF (g/pot)} = [\% \text{NSPF}/100] \times \text{Np}$ , where: Np = accumulated N in the soil from the pots (g);
4. Recovery of N applied to the soil  
 $\text{Rec} = (\text{ASNFF}/\text{QAN}) \times 100$ , where: QAN = quantity of N applied
5. Volatilization losses  
 $\text{Losses} = 100 - \text{Rec}$ .

Data were analyzed by ANOVA and means compared by Tukey's test using SAS software (SAS, 1999). For accumulated  $\text{NH}_3\text{-N}$  volatilization the reference method was the  $^{15}\text{N}$  balance method. For  $\text{NH}_3\text{-N}$  daily volatilization rate, daily values were compared using values from the semi-open static collector method as reference.



## RESULTS AND DISCUSSION

Estimates of accumulated loss of volatilized NH<sub>3</sub>-N based on the semi-open static collector were similar only to estimates obtained by foam absorber 1 cm (FA1) and <sup>15</sup>N balance (<sup>15</sup>N) methods ( $p < 0.01$ ). No calibration equation or correction factor was necessary to estimate the accumulated loss of volatilized NH<sub>3</sub>, as occurred with the results obtained by Araújo et al. (2009) and Lara Cabezas et al. (1999). The use of acid absorbers 1, 5 and 10 cm (AA1, AA5 and AA10) and paper absorbers 1 and 5 cm (PA1 and PA5) above the soil resulted in similar values for accumulated NH<sub>3</sub>-N compared to <sup>15</sup>N balance ( $p < 0.01$ ) (Table 1). Therefore, the semi-open collector, FA1, AA1, AA5, AA10, PA1, and PA5 estimated the real accumulated NH<sub>3</sub>-N losses. Manzoni & Trivelin (personal communication) compared the paper absorber and the <sup>15</sup>N balance methods observing that the paper absorber 1 cm only estimated real NH<sub>3</sub>-N losses by volatilization when volatilization was high.

Foam absorbers at 5, 10 and 20 cm (FA5, FA10 and FA20) and the paper absorbers 10 and 20 cm (PA10 and PA20) underestimated N-NH<sub>3</sub> volatilization losses. It is possible that part of the volatilized NH<sub>3</sub>-N was dispersed in the air before reaching the absorbers.

The estimates of accumulated NH<sub>3</sub>-N losses were similar for FA1, AA1, AA5, AA10, PA1, PA5 and <sup>15</sup>N balance methods. However, in order to verify if these methods were able to estimate the real NH<sub>3</sub>-N volatilization, it would be necessary to compare not only the accumulated values, but the values of the daily NH<sub>3</sub>-N loss rate with the reference method (<sup>15</sup>N balance), that is, with the curve of ammonia volatilization rates over time. NH<sub>3</sub>-N volatilization by the <sup>15</sup>N balance method was only evaluated on the last day of the experimental period, so an estimation

of the daily rate of ammonia loss by this method was not possible. Nevertheless, the semi-open collector had already been calibrated by the <sup>15</sup>N balance method by Lara Cabezas et al. (1999). Consequently, the NH<sub>3</sub>-N loss values obtained by the semi-open collector were compared statistically with values from the other absorbers for each evaluation day (Figures 3, 4 and 5).

From the 4<sup>th</sup> to the 10<sup>th</sup> day after urea application the losses estimated by FA1 were similar to those estimated by the semi-open collector (Figure 3). Losses are considered more relevant during this period. On the 14<sup>th</sup> and 22<sup>nd</sup> day, losses estimated by both methods were equal again ( $p < 0.01$ ).

With FA1 it was possible to obtain the real estimates of the accumulated NH<sub>3</sub>-N loss by volatilization and the daily rate ammonia loss by volatilization.

Although curves for results by FA5, FA10 and FA20 methods indicated lower NH<sub>3</sub>-N volatilization values when compared to the semi-open collector, the pattern was the same, with a peak for NH<sub>3</sub>-N loss on the sixth day. The similarity of these curves indicated that the calibration of these methods with <sup>15</sup>N may be possible, as similarly done in the case of the semi-open collector by Lara Cabezas & Trivelin (1999) and Araújo et al., 2009.

Losses estimated by PA1 and PA5 were the same as by the semi-open collector, from the 6<sup>th</sup> to the 10<sup>th</sup> day and on the 14<sup>th</sup> and 22<sup>nd</sup> day after urea application (Figure 4).

On the 6<sup>th</sup>, 10<sup>th</sup> and from the 14<sup>th</sup> to the 18<sup>th</sup> day after urea application, the amount of volatilized NH<sub>3</sub>-N estimated by the semi-open collector method was equal to the AA1 method. Volatilization estimated by AA5 and the semi-open collector was similar from the 8<sup>th</sup> to the 10<sup>th</sup> day and from the 14<sup>th</sup> to the 18<sup>th</sup> day after urea application (Figure 5). Ammonia

**Table 1. Accumulated losses of volatilized NH<sub>3</sub>-N estimated by the tested experimental methods**

Treatment		Accumulated losses of volatilized N-NH <sub>3</sub>
Method	Height above the soil surface	
	cm	mg/plot
Semi-open static collector	-	69.88 a
Foam absorber	1	53.89 ab
<sup>15</sup> N balance (reference method)	-	46.95 abc
Acid absorber	1	44.01 bcd
Acid absorber	5	35.53 bcde
Acid absorber	10	34.03 bcde
Paper absorber	1	27.88 cde
Paper absorber	5	25.87 cde
Foam absorber	10	21.96 de
Foam absorber	5	21.32 de
Paper absorber	10	13.33 e
Foam absorber	20	13.33 e
Paper absorber	20	12.14 e
CV (%)	-	34.70

Means followed by different letters are different ( $P \leq 0.01$ ) by Turkey's test.

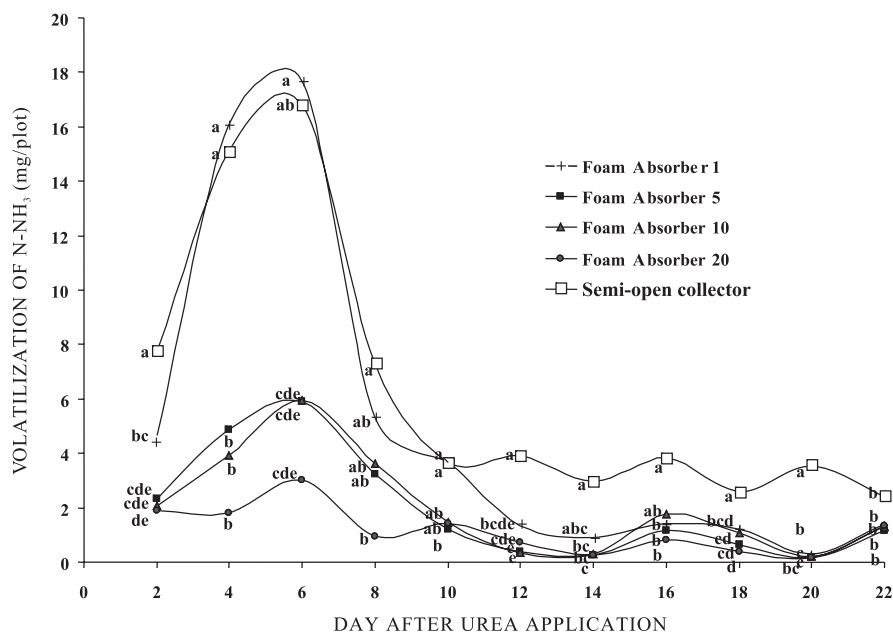


Figure 3. Estimates of volatilized  $\text{NH}_3\text{-N}$  losses by the semi-open collector (CSA), foam absorber at 1 cm, foam absorber at 5 cm, foam absorber at 10 cm and foam absorber at 20 cm. <sup>a,b,c,d</sup> Equal letters indicate equal means by Tukey's test at 1%. (Coefficient of Variation % day 2 = 43.90; day 4 = 54.54; day 6 = 51.56; day 8 = 60.10; day 10 = 48.56; day 12 = 41.62; day 14 = 97.65; day 16 = 67.38; day 18 = 40.86; day 20 = 74.81; day 22 = 35.48).

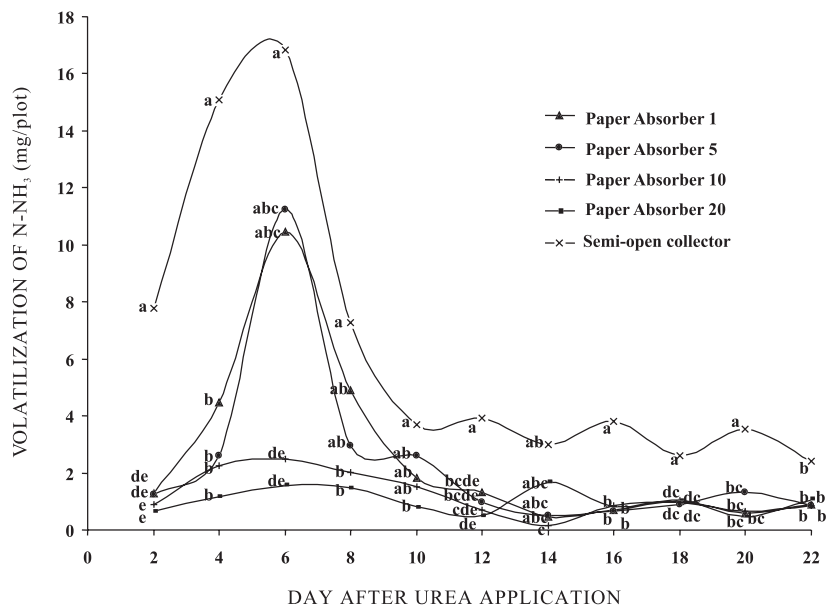


Figure 4. Estimates of volatilized  $\text{N-NH}_3$  losses by the semi-open collector (CSA), paper absorber at 1 cm, at 5 cm, at 10 cm and at 20 cm. <sup>a,b,c,d</sup> Equal letters indicate equal means by Tukey's test at 1%. Coefficient of Variation (%) on day 2 = 43.90; day 4 = 54.54; day 6 = 51.56; day 8 = 60.10; day 10 = 48.56; day 12 = 41.62; day 14 = 97.65; day 16 = 67.38; day 18 = 40.86; day 20 = 74.81; day 22 = 35.48).

volatilization reached maximum values between the 4<sup>th</sup> and 6<sup>th</sup> day after urea application, as reported by Lara Cabezas & Trivelin (1990) and Costa et al. (2003).

It was observed that the amounts of lost and captured  $\text{NH}_3\text{-N}$  detected by FA1 were most similar to those of the semi-open collector, in the 22 days of

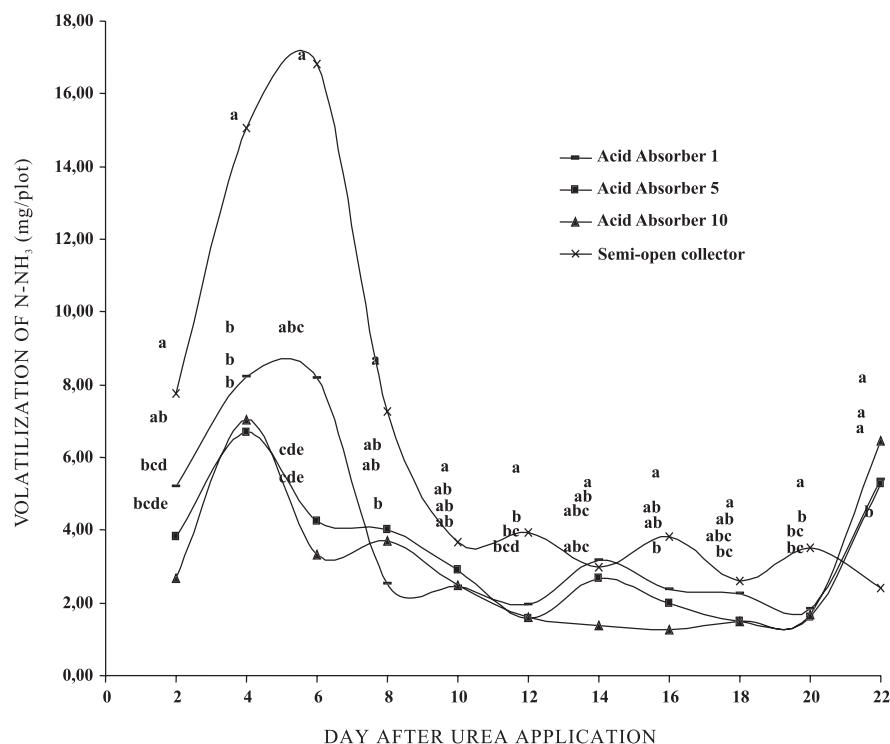


Figure 5. Estimates of volatilized  $\text{N-NH}_3$  losses by the semi-open collector (CSA), acid absorber at 1 cm, at 5 cm and at 10 cm. <sup>a,b,c,d</sup>Equal letters indicate equal means by Tukey's test at 1%. (Coefficient of Variation (%) on day 2 = 43.90; day 4 = 54.54; day 6 = 51.56; day 8 = 60.10; day 10 = 48.56; day 12 = 41.62; day 14 = 97.65; day 16 = 67.38; day 18 = 40.86; day 20 = 74.81; day 22 = 35.48).

evaluation. Since estimates of  $\text{NH}_3\text{-N}$  volatilization by FA1 and semi-open collector were equal to estimates by the  $^{15}\text{N}$  balance method, it is possible to indicate FA1 as an advantageous alternative to the other two methods, mainly because it is less expensive and does not interfere with the natural process of  $\text{NH}_3$  volatilization from the soil either, as verified by Lara Cabezas & Trivelin (1990) for the semi-open collector.

The evaluation of the FA1 method under field conditions is under way, with a view to quantify ammonia volatilization and foliar emission in pastures.

## CONCLUSION

Polyurethane foam with phosphoric acid solution absorber (foam absorber) installed 1 cm above the soil surface is efficient in capturing  $\text{N-NH}_3$  volatilized from urea-treated soil, estimating the daily and accumulated loss rates of  $\text{N-NH}_3$  correctly.

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